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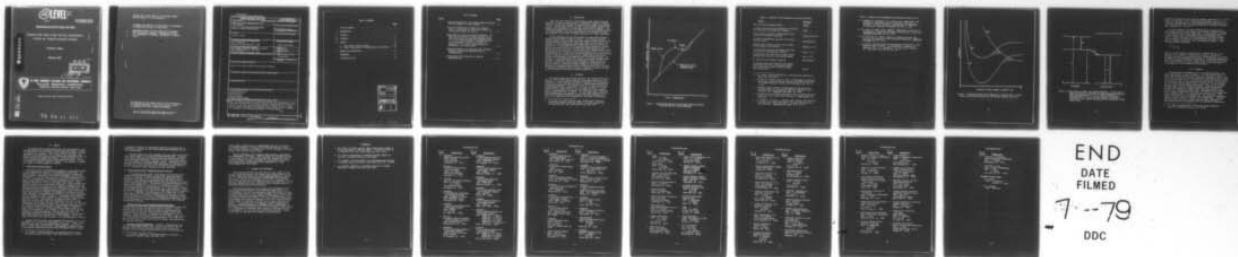
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MEMORANDUM REPORT ARBRL-MR-02902

SEARCH FOR LOW-LYING TRIPLET ELECTRONIC
STATES OF SIMPLE NITRATE ESTERS

Richard A. Beyer

February 1979

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I. INTRODUCTION

Since the first reports¹ of the effects of lead compounds on the ballistics of double base propellants a substantial amount of research has been occupied with attempts to understand the causes of the observed effects. The greatest interest has been by rocket engine designers who benefit from the presence of the plateau region shown in Figure 1. Gun propellant designers have also been interested in this phenomenon because of the modification of ignition properties of these materials as well the extension of the effect to higher pressures or to other propellants.

Several explanations have been proposed for super-rate burning; a summary of these ideas is given in Table I. Of particular interest is the possible role of excited electronic states of nitrocellulose (NC). Although the work reported here deals with simple nitrate esters, the energies of the low-lying electronic states should be very similar to those of NC. If one considers the nitrate ester decomposition products, NO_2 and an alkoxyl radical, it is found that these species in their ground states correlate with a triplet state of the nitrate ester. Since the nitrate ester is clearly in the ground singlet state at the start of decomposition, any process which can mix the singlet and low-lying triplet states or populate a triplet state in any manner would substantially increase the unimolecular decomposition rate of the nitrate ester. An increase in the decomposition could result in the faster burning rates observed. A schematic diagram of the electronic states involved in the above process as well as possible pathways of decomposition are shown in Figure 2. The goal of these studies was to determine the energies of the lowest triplet states of simple nitrate esters in order to determine whether they are low enough in energy to interact with the ground electronic states during thermal decomposition.

II. APPROACH

The lowest energy bound triplet states of the simple nitrate esters have not been observed spectroscopically since the transition is spin forbidden. A direct method for showing the presence of an unobservable triplet state is to show that the state in question makes the molecule an efficient quencher of the phosphorescence of a second molecule with a higher-energy lowest triplet state. As shown in Figure 3, a donor molecule with a triplet state T_1 of known energy and a good quantum yield of phosphorescence is selected. The exciting light is absorbed, causing population of S_1 via path a. For many large organic molecules rapid intramolecular intersystem crossing via path b populates the state T_1 in competition with re-emission of the light (fluorescence).

1. W.H. Avery, R.E. Hunt, and M.N. Down, "Burning Rate Studies In Double Base Powders", OSRD 5827 ABL/P/1, Jan 1946 as quoted by H. Singh and K.R.K. Rao, *AIAA Journal*, 15, 1545 (1977).

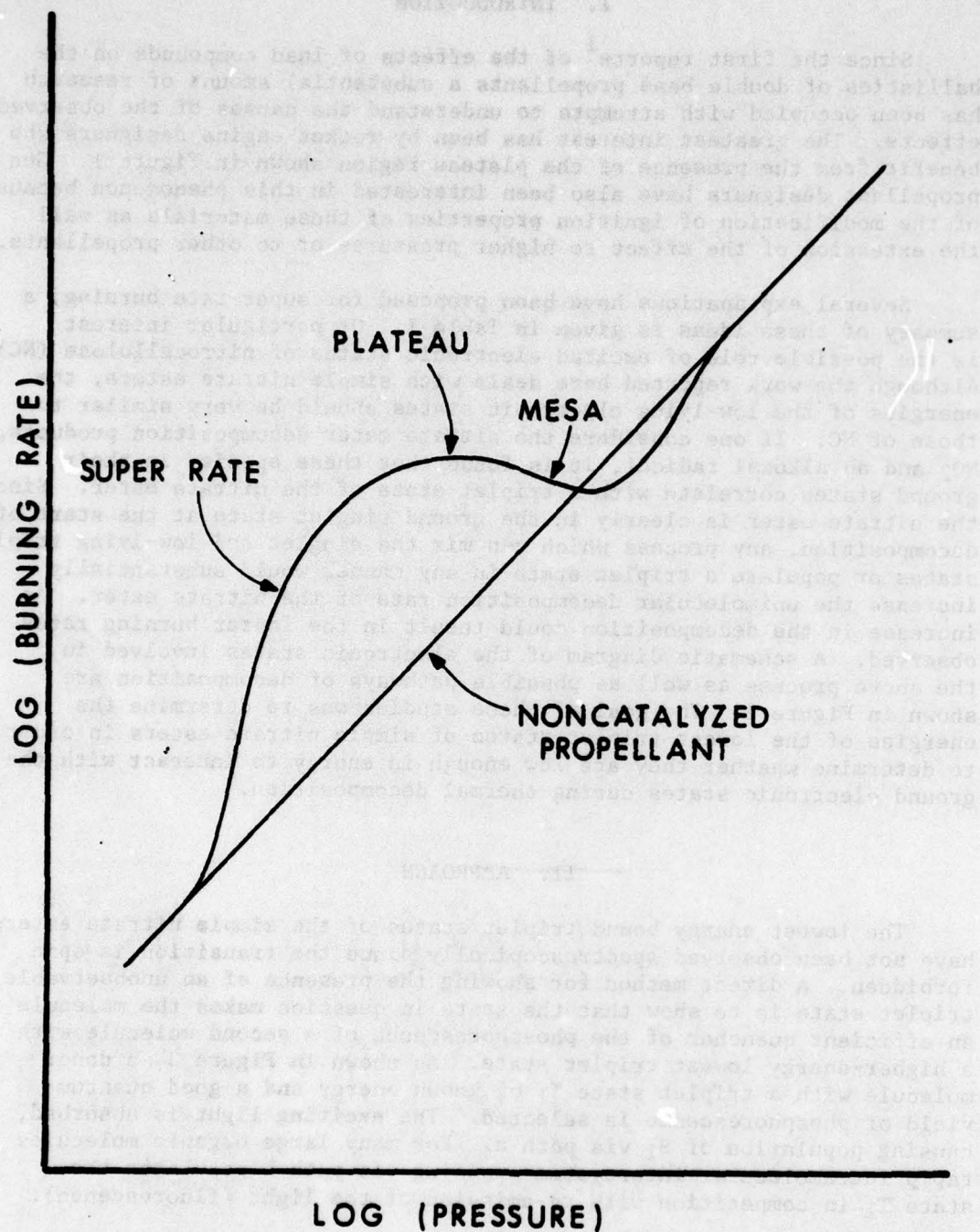


Figure 1. Idealized Burning Rate Curve Showing Regions Obtained with the Addition of Lead Compounds.

TABLE I. Theories of the Mechanism of Super-Rate Burning

Theory	Reference
PbO reacts with nitrogen oxides.	Prekel ^a
Pb below the burning surface absorbs Uv radiation from the flame to increase heat feedback.	Camp ^b
More energetic reactions (unspecified) at the surface of a modified propellant.	Lenchitz/Haywood ^c
Pb affects aldehyde/NO ₂ reactions in the foam and fizz zones.	Heller/Gordon ^d
Surface carbon reacts with NO ₂ ; PbO is cross-linking agent to form carbon.	Hewkins, et al ^e
Increased NO ₂ production accelerates fizz zone reactions and increases heat feedback.	Kubota, et al ^f
Lead modifier acts chemically to speed initial decomposition reaction in sub-surface region.	Suh, et al ^g
Pb catalyzes CO ₂ formation reactions.	Fifer/Lannon ^h
Pb enhances spin-orbit coupling of the ground electronic state with a dissociative triplet state resulting in lower activation energy for decomposition.	Harris ⁱ

a - R.F. Prekel, "Plateau Ballistics in Nitrocellulose Propellants", ARS J., 31, 1286 (1961).

b - L.A. Lee, T.D. Austin, and A.T. Camp, "A Photochemical Combustion Mechanism for Mesa and Plateau Burning Double-Base Propellants", 1974 JANNAF Combustion Meeting, CPIA Publication 262, Vol. 1, p. 293.

c - C. Lenchitz and B. Haywood, "Determination of the Role of the Ballistic Modifier in Propellant Combustion Using the Heat of Explosion Test", Combustion and Flame, 10, (1966).

d - C.A. Heller and A.S. Gordon, Structure of the Gas Phase Combustion Region of a Solid Double Base Propellant", J. Phys. Chem., 59, 773 (1955).

e - D.J. Hewkins, J.A. Hieks, J. Pawling, and H. Watts, "The Combustion of Nitric Ester-Based Propellants: Ballistic Modification by Lead Compounds", Combustion Sci. Tech., 2 307 (1971).

TABLE I. Theories of the Mechanism of Super-Rate Burning (Cont'd)

- f - K. Kubota, T.J. Ohlemiller, L.H. Caveny, and M. Summerfield, "The Mechanism of Super-Rate Burning of Catalyzed Double Base Propellants", Fifteenth Symposium (International) on Combustion, The Combustion Institute, Pittsburgh, PA, 1974, p. 529.
- g - N.P. Suh, G.F. Adams, and C. Lenchitz, "Observation on the Role of Lead Modifier in Super-Rate Burning of Nitrocellulose Propellants", Combustion and Flame, 22, 289 (1974).
- h - R.A. Fifer and J.A. Lannon, "Effects of Pressure and Some Lead Salts on the Chemistry of Solid Propellant Combustion", Combustion and Flame, 24, 369 (1975).
- i - L.E. Harris, "The Reactivity of Nitrate Esters" published in "Army Materiel Command Program: The Fundamentals of Ignition and Combustion; Vol. II: Combustion", I.W. May and A.W. Barrows, eds., BRL Report 1708, AD# 919316L, 1974.

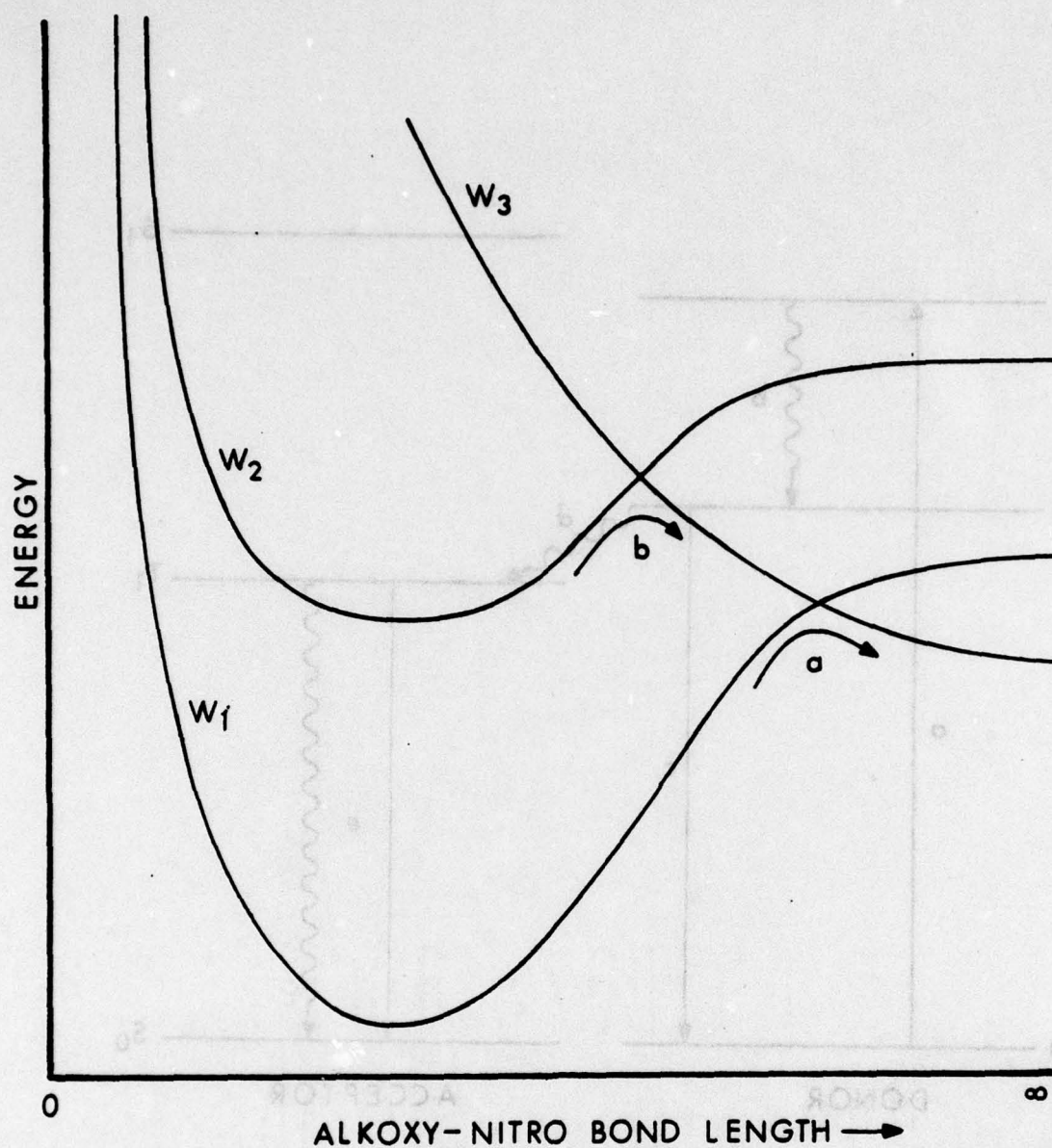


Figure 2. Electronic Energy Levels Diagram for a Nitrate Ester. States Shown are the Ground Singlet, W_1 , and Excited Triplets, W_2 and W_3 .

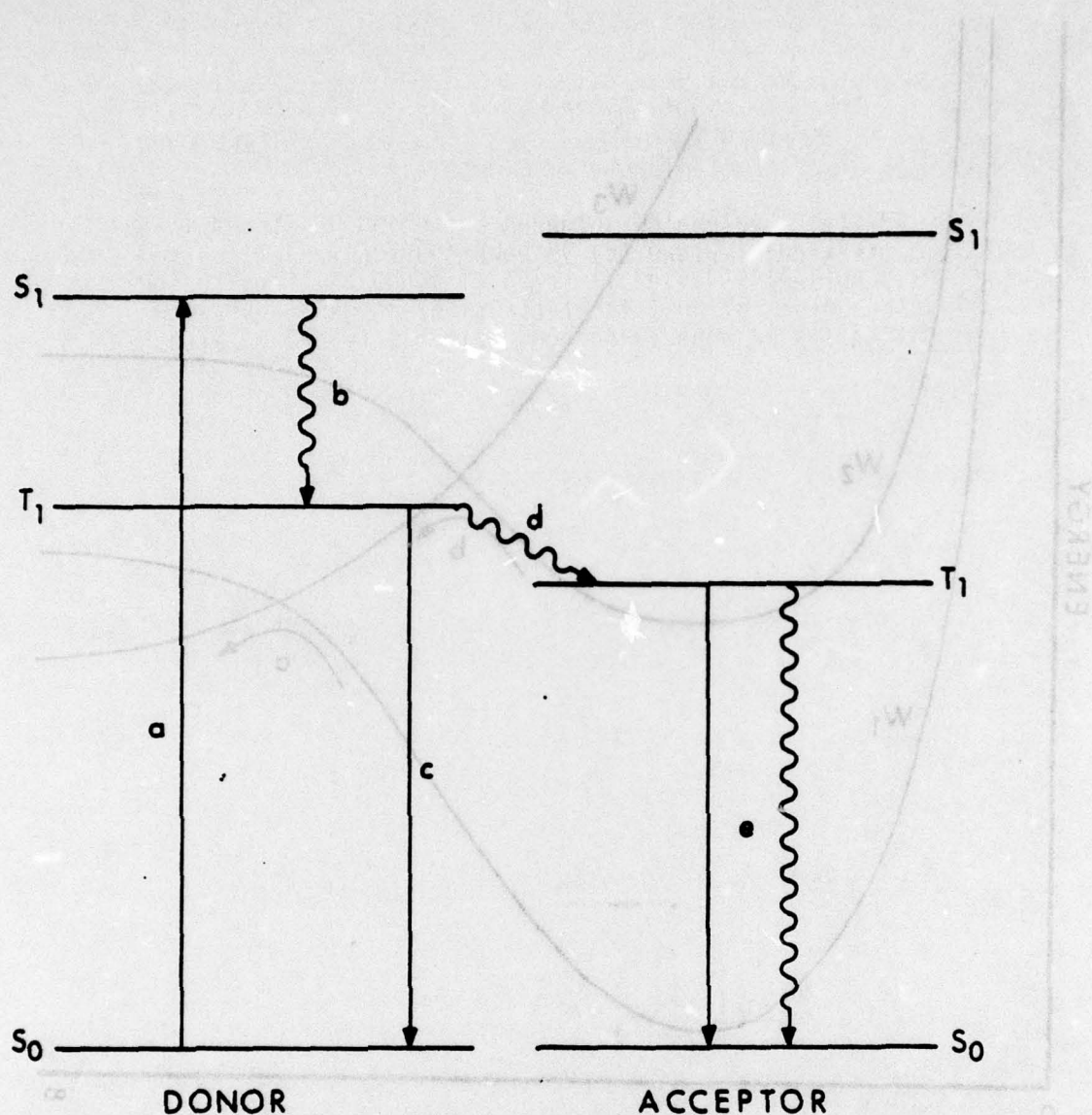


Figure 3. Energy Levels of Donor and Acceptor Molecules. (a) The Donor is Optically Excited, Followed by (b) Rapid Intersystem Crossing Which Populates Donor T_1 . (c) T_1 Loses Excitation via Phosphorescence or (d) Collisional Transfer to Acceptor T_1 , Resulting in (e) Acceptor Phosphorescence or Relaxation to the Ground State.

Emission from either excited state has intensity described by $I = I_0 e^{-t/\tau}$, where t is time and τ is the characteristic lifetime. As the $T_1 \rightarrow S_0$ transition (phosphorescence) is spin-forbidden and allowed only due to spin-orbit perturbations, the light emitted has a relatively long lifetime, typically 10^{-4} to 10^{-2} s.

If one introduces an acceptor molecule with a triplet state of approximately equal or lower energy, very efficient transfer of energy to the acceptor triplet state takes place. If the acceptor state has a long lifetime, then the quenching of the phosphorescence of the donor is readily observable by an attenuation of the donor lifetime with the degree of attenuation of the lifetime proportional to the number of acceptor molecules. Thus, the measured lifetime, τ , obeys the Stern-Volmer relationship,

$$\frac{1}{\tau} = \frac{1}{\tau_0} + k_q$$

where τ_0 is the lifetime in the absence of quenching and k_q is the average rate of transfer of energy from the donor. One may also measure a decrease in the quantum yield of phosphorescence of the donor, as evidence of the quenching by the acceptor. It has been demonstrated that this quenching is extremely efficient and limited only by mobility of the species when the acceptor triplet level is more than 1000 cm^{-1} (3 kcal/mole or 0.02 eV) below that of the donor.

III. APPARATUS

The experimental arrangement used for lifetime measurements and other observations is shown in Figure 4. The light source used was a 500 W high-pressure Hg vapor lamp chopped by a mechanical chopper. The light pulse frequency and duration were varied according to the lifetime of the molecule under study. Colored glass filters were used to isolate the excitation light to the appropriate spectral region for absorption into the lowest excited singlet state of the donor. The pyrex sample cell was fitted with UV grade quartz windows. Emission was focussed through either a variable wedge interference filter monochrometer, colored glass filters, or a small quartz prism monochrometer, and detected by a 1P28 photomultiplier tube. The signals were recorded using a signal averager with time resolution down to 20 μs /channel with 9 bit resolution and to 1 μs /channel with 6 bit resolution. Under typical conditions data were averaged for a few minutes to obtain good signal to noise, as shown in Figure 5 for biacetyl decay. Averaged data were then punched out on paper tape for further analysis or photographed from an oscilloscope trace for qualitative observations.

2. A.A. Lamola, "Applications of Electronic Energy Transfer in Solution", *Photochem Photobiol* 8, 601 (1968).

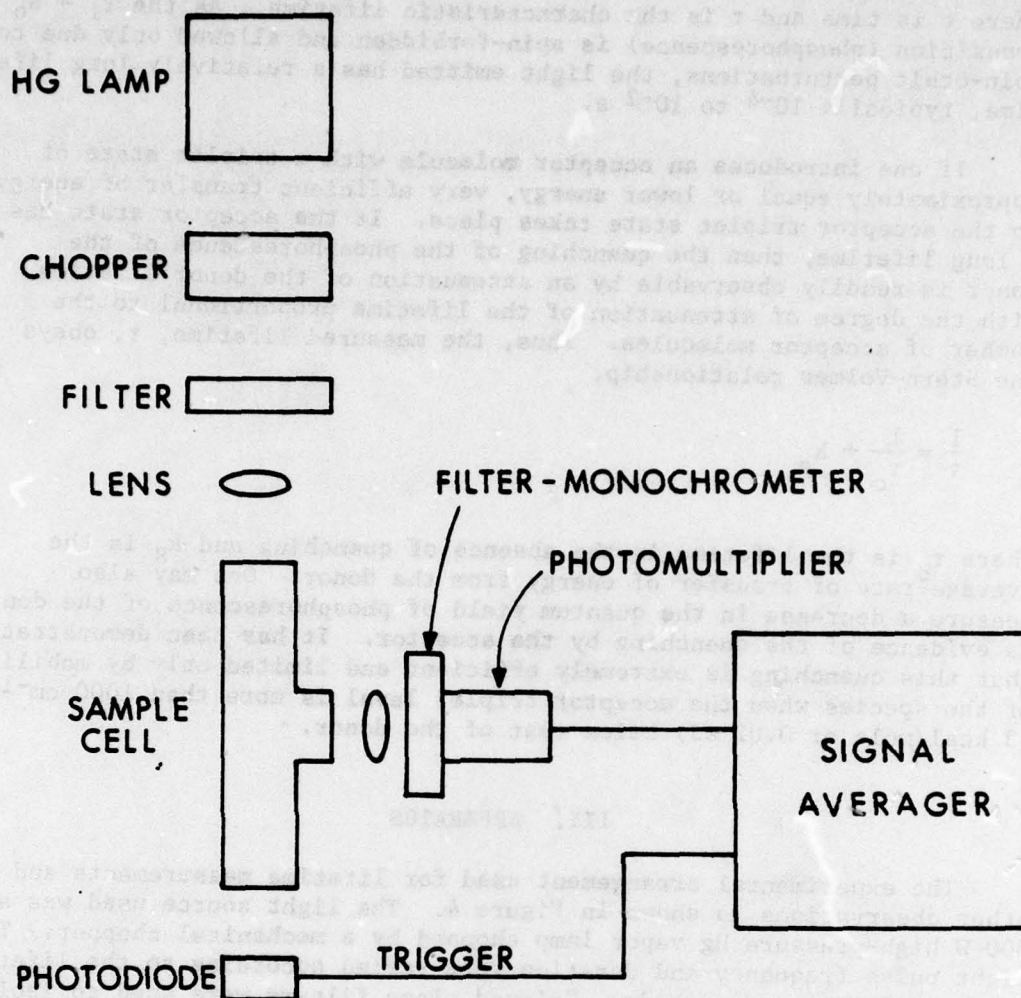


Figure 4. Schematic Diagram of the Apparatus Used to Measure Lifetimes and Relative Quantum Yields of Phosphorescence.

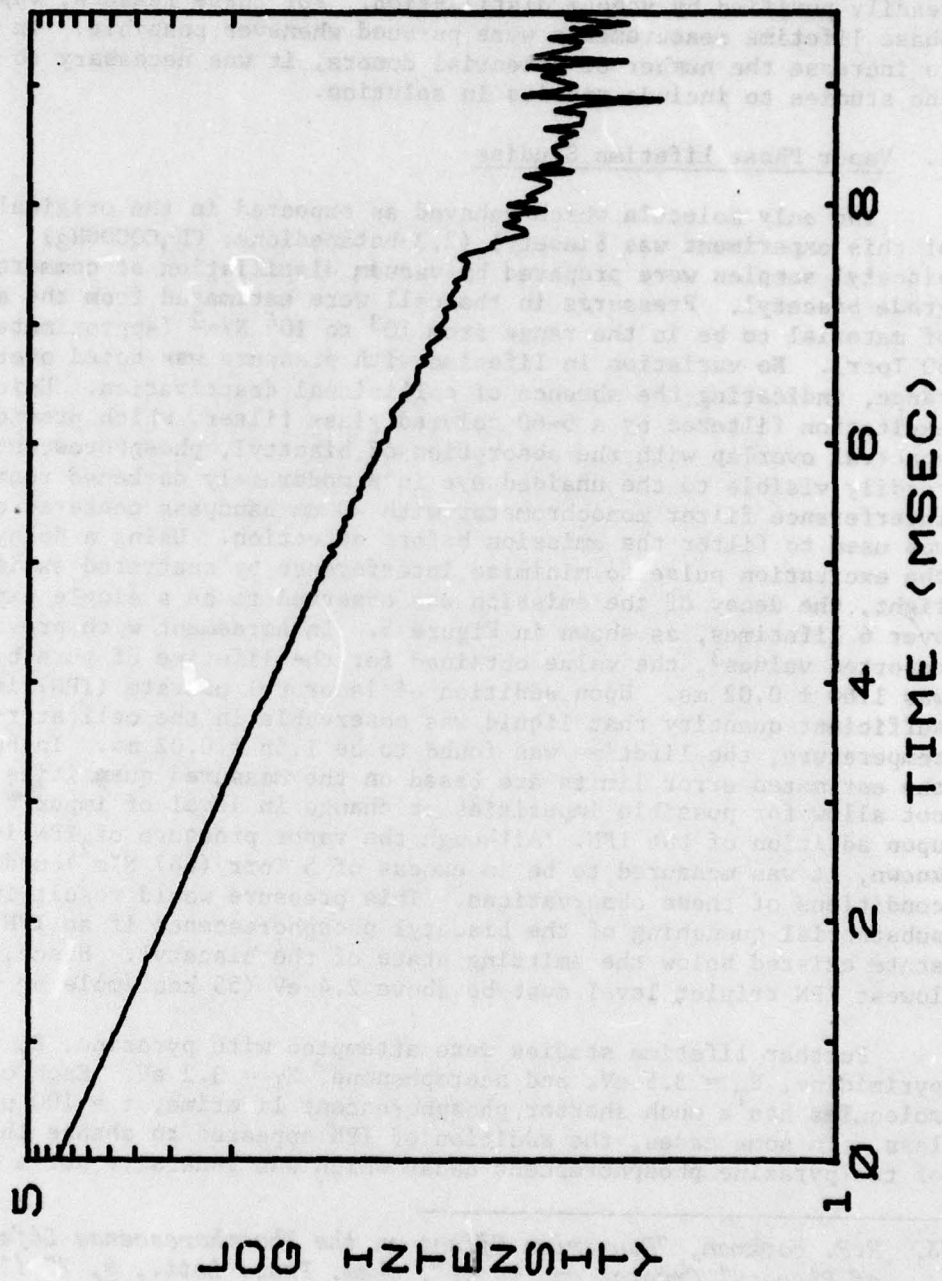


Figure 5. Typical Decay Curve Recorded for Biacetyl Phosphorescence.

IV. RESULTS

The observations and measurements fall into two categories, lifetime measurements and qualitative quantum yield measurements. Because of elimination of solvent effects on the lifetime, gas phase lifetime measurements are preferred. Also, materials with sufficient vapor pressure at room temperature to allow vapor phase measurements are more readily purified by vacuum distillation. For these reasons, vapor phase lifetime measurements were pursued whenever possible. In order to increase the number of potential donors, it was necessary to expand the studies to include species in solution.

A. Vapor Phase Lifetime Studies

The only molecule which behaved as expected in the original design of this experiment was biacetyl (2,3-butanedione; $\text{CH}_3\text{COCOCH}_3$). Pure biacetyl samples were prepared by vacuum distillation of commercial grade biacetyl. Pressures in the cell were estimated from the amount of material to be in the range from 10^3 to 10^4 N/m² (approximately 5 to 50 Torr). No variation in lifetime with pressure was noted over this range, indicating the absence of collisional deactivation. Using excitation filtered by a 5-60 colored glass filter, which provides good spectral overlap with the absorption of biacetyl, phosphorescence was readily visible to the unaided eye in a moderately darkened room. The interference filter monochromator with 40 nm bandpass centered at 520 nm was used to filter the emission before detection. Using a delay after the excitation pulse to minimize interference by scattered excitation light, the decay of the emission was observed to be a single exponential over 6 lifetimes, as shown in Figure 5. In agreement with previously reported values³, the value obtained for the lifetime of pure biacetyl was 1.60 ± 0.02 ms. Upon addition of isopropyl nitrate (IPN) in sufficient quantity that liquid was observable in the cell at room temperature, the lifetime was found to be 1.66 ± 0.02 ms. In both cases, the estimated error limits are based on the measured quantities but do not allow for possible impurities or change in level of impurities upon addition of the IPN. Although the vapor pressure of IPN is not known, it was measured to be in excess of 5 Torr (667 N/m²) under the conditions of these observations. This pressure would result in substantial quenching of the biacetyl phosphorescence if an IPN triplet state existed below the emitting state of the biacetyl. Hence, the lowest IPN triplet level must be above 2.4 eV (55 kcal/mole or 0.38 aJ).

Further lifetime studies were attempted with pyrazine, $E_T = 3.3$ eV, pyrimidine, $E_T = 3.5$ eV, and acetophenone, $E_T = 3.2$ eV. Each of these molecules has a much shorter phosphorescent lifetime, $\tau = 100$ μ s or less. In some cases, the addition of IPN appeared to change the shape of the pyrazine phosphorescent decay which was generally not a simple

3. R.F. Borkman, "Deuterium Effect on the Phosphorescence Lifetime of Biacetyl Crystal and Vapor", *Chem. Phys. Lett.*, **9**, 77 (1971).

exponential. However, no reproducible effects were observed and no quenching was observed for the other species whose emissions were not well characterized.

Lifetime studies were also attempted with vapor phase benzaldehyde with results similar to the three species mentioned above. Scattered light from an apparent photochemical product was observed and found to increase with irradiation. Addition of IPN to this system did not slow the formation of the photochemical product. Although it would be expected that the photochemistry might take place via a triplet state, one cannot be certain that the path is through the lowest triplet at 3.1 eV. Therefore, these observations are not conclusive.

B. Visual Observations of Phosphorescence in Solution

As the apparent lower limit of the energy of the nitrate ester triplet state was pushed upward, suitable donor molecules became difficult to propose. It was noted that some compounds are seen to phosphoresce in solution at room temperature; this effect makes it possible to make qualitative observations which may bracket the nitrate ester triplet energy level. The presence of substantial phosphorescence implies that quenching by the solvent (or impurities) either via intersystem crossing to the ground state or by triplet-triplet transfer is slow compared with the phosphorescence rate. The lifetimes of these emissions was observed to be too fast for measurements with our apparatus ($\tau < 1 \mu s$). Quenching of phosphorescence by a solvent with a lower triplet state than the origin of emission should take place on a very short time scale, 10 ps or faster, eliminating any observable emission if the triplet state of the solvent is effectively non-emitting. Using the highest grades of solvents available commercially, the following observations were made.

Michler's Ketone (p,p'-bisdimethylaminobenzophenone).

Phosphorescence followed the shortest available excitation pulse using acetone, acetonitrile, and ethylene glycol as solvents. A green emission which is most certainly the expected phosphorescence was clearly visible to the eye. Using IPN and n-propyl nitrate (NPN) separately as the solvents, the emission was unchanged in color or intensity. These observations indicate that the lowest triplet state of IPN and NPN must be above the emitting state at 2.6 eV (60 kcal/mole or 0.49 aJ); if either were below this energy, the emission would be completely quenched.

Fluorene (diphenylenemethane). As above, a green emission was observed using either acetone, IPN, or NPN as the solvent, and the emission again followed the fastest available excitation pulse. Fluorene is a well characterized molecule⁴, and it is known that its

4. I.B. Berlman, "Handbook of Fluorescence Spectra of Aromatic Molecules", Academic Press, New York, 1965.

lowest energy fluorescence is at approximately 360 nm or far blue of the observed emission. As above, we can conclude that the nitrate ester triplet states must be above the 2.9 eV (67 kcal/mole or 0.55 aJ) energy of the fluorene triplet state.

Similar experiments were attempted using pyrazine, benzophenone, and benzaldehyde in solution. With the latter two, emission was weak or not present at all. With pyrazine some blue emission was clearly observed; however, attempts to separate it from scattered excitation light using either colored glass filter combinations or simple monochrometers were unsuccessful.

V. SUMMARY AND CONCLUSIONS

Three observations have been made that set lower limits for the vertical transition energy to the lowest triplet energy levels of IPN and NPN. These observations are the lifetime measurements of biacetyl ($E_T = 2.4$ eV) and the non-quenching of phosphorescence of Michler's ketone ($E_T = 2.6$ eV) and of fluorene ($E_T = 2.9$ eV). Although there were also indications that the phosphorescence of pyrazine ($E_T = 3.3$ eV) was being affected under some conditions, purity problems prevented a definitive observation with this molecule.

From these observations we can conclude that the energy of the lowest-lying triplet states of IPN and NPN are higher than 2.9 eV (67 kcal/mole or 0.55 aJ) above the ground state and probably too high to be important directly in the thermal decomposition of nitrate esters. A closely related and unanswered question of the possible role of the dissociative triplet state remains. This state could be mixed with the ground singlet via spin-orbit coupling to allow for a lower activation energy channel for breaking the RO-NO₂ bond as shown in pathway a of Figure 2. If important, this collisionally induced predissociation should be readily observable as an enhanced rate of dissociation in the presence of heavy molecules such as Xe as compared to lighter gases. If no enhancement is observable under laboratory conditions it would be reasonable to rule out spin-orbit perturbations as being a significant factor in the super-rate burning of nitrocellulose.

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4. I.B. Berlman, "Handbook of Fluorescence Spectra of Aromatic Molecules", Academic Press, New York, 1965.

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